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Applicant: UNILEVER PLC Unilever House Blackfriars P.O. Box 68 London EC4P 4BQ(GB)

⊕ GB

Applicant: UNILEVER NV
Burgemeester s'Jacobplein 1 P.O. Box 760
NL-3000 DK Rotterdam(NL)

(A) CH DE ES FR IT LI NL SE

② Inventor: Barber, Alan Dan, Unilever Research Port Sunlight Lab., Quarry Road East, Bebington
Wirral, Merseyside, L63 3JW(GB)
Inventor: Emery, William Derek, Unilever Research
Port Sunlight Lab., Quarry Road East, Bebington
Wirral, Merseyside, L63 3JW(GB)

Representative: Fransella, Mary Evelyn et al Unilever PLC Patents Division P.O. Box 68 Unilever House London EC4P 4BQ(GB)

Detergent compositions.

A granular detergent base composition having a bulk density of at least 650 g/litre comprises:
 an organic surfactant system consisting essentially of fatty acid soap (5-40 wt% on composition), alkoxylated nonionic surfactant (5-35 wt% on composition) and preferably free of nonsoap anionic surfactant; a detergency builder system consisting essentially of alkali metal aluminosilicate (20-60 wt% anhydrous on composition) and an organic sequestrant builder such as alkali metal citrate (2-15 wt% on composition); and optionally various other detergent ingredients such as sodium carbonate, sodium silicate, sodium succinate.

The detergent base composition can be prepared by densification of a spray-dried base powder in a high-speed mixer/granulator. It combines good detergency with a lack of environmentally questionable ingredients, and dispenses well in a drum-type washing machine despite the high bulk density.

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DETERGENT COMPOSITIONS

TECHNICAL FIELD

The present invention relates to a free-flowing granular detergent base composition of high bulk density having a surfactant system consisting essentially of fatty acid soap and nonionic surfactant, and a detergency builder system based on aluminosilicate. The composition of the invention, which can be used alone or admixed with other detergent ingredients, combines efficient cleaning performance with a lack of environmentally questionable ingredients, has good powder properties, and dispenses surprisingly well into a drum-type automatic washing machine.

The invention also relates to the preparation of the detergent base composition by densification of a spray-dried base powder in a high-speed mixer/granulator. In one embodiment in which a bowl-type batch mixer is used, a product having an exceptionally attractive appearance is obtained.

15 PRIOR ART

EP 344 629A (Henkel), published on 6 December 1989, discloses a granular composition comprising aluminosilicate (45-75 wt%), fatty acid soap (1-6 wt%), acrylic and/or maleic polymer (1-12 wt%), optionally plus sodium sulphate and a small amount (up to 5 wt%) of nonionic surfactant. The granular material (65-90 wt%) may be used as a carrier for nonionic surfactant (10-35 wt%).

EP 229 671A (Kao), JP 61 069 897A (Kao) and JP 61 064 798A (Kao) disclose the densification of spray-dried detergent powder, containing a high level of anionic non-soap surfactant and a relatively low level of aluminosilicate builder, in a high-speed mixer/granulator.

EP 340 013A (Unilever), published on 2 November 1989, describes and claims high-bulk-density granular detergent compositions comprising moderate levels of anionic non-soap surfactant and moderate levels of aluminosilicate builder, and their preparation by densification of spray-dried base powder in a high-speed mixer/granulator.

JP 62 086 099A (Nippon Oils & Fats) discloses a granular soap-based detergent composition of high bulk density typically containing 40-55 wt% soap, 5-20 wt% nonionic surfactant (7-15 wt% exemplified) and 25-50 wt% builder. The powder is prepared by a non-tower process in which soap chips, nonionic surfactant and builders are disintegrated and mixed in a lateral-type mixer/granulator.

DEFINITION OF THE INVENTION

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The present invention provides a granular detergent base composition having a bulk density of at least 650 g/litre, which comprises:

- (i) an organic surfactant system consisting essentially of:
 - (a) from 5 to 40 wt% of fatty acid soap,
 - (b) from 5 to 35 wt% of alkoxylated nonionic surfactant;
- (ii) a detergency builder system consisting essentially of:
 - (c) from 20 to 60 wt% (anhydrous basis) of crystalline or amorphous alkali metal aluminosilicate,
 - (d) from 2 to 15 wt% of an organic sequestrant builder;
- (iii) optionally from 0 to 25 wt% sodium carbonate, with the proviso that if the content of fatty acid soap is ≤6 wt%, from 2 to 25 wt% sodium carbonate is present;
- (iv) optionally other detergent ingredients and moisture 30 to 100 wt%, all percentages being based on the detergent base composition.

The invention also provides a process for the preparation of a detergent base composition as defined in the previous paragraph, which includes the steps of:

- (1) spray-drying an aqueous slurry to form a base powder, and
- (2) densifying the spray-dried base powder in a high-speed mixer/granulator having both a stirring action and a cutting action.

DETAILED DESCRIPTION OF THE INVENTION

The detergent base composition

The first subject of the present invention is a granular detergent base composition of moderate to high bulk density, having a surfactant system based on fatty acid soap and alkoxylated nonionic surfactant, and built with alkali metal aluminosilicate, at a relatively high level, and an organic sequestrant cobuilder.

The term "detergent base composition" is intended to indicate a particulate composition that may be used as a detergent product in its own right, or alternatively may be admixed with other, separately prepared ingredients to form a more fully formulated detergent composition. The detergent base composition is a generally a substantially homogeneous particulate material.

The invention is especially concerned with a spray-dried detergent base composition, which may or may not be subsequently densified. Compositions of the invention having bulk densities up to about 650 g/l have been successfully produced just by spray-drying, and these may be further densified by post-tower treatment in a high-speed mixer/granulator as indicated above.

In the specific context of spray-drying, the term "detergent base composition" indicates the spray-dried primary product, plus any "base" ingredients (for example, nonionic surfactant, fatty acid soap) that for processing reasons may be withheld from the slurry and added after the tower.

For example, as described below under "Process for preparation of the detergent base composition", it is within the scope of the invention for the liquid or liquefiable ingredients of the base composition -nonionic surfactant, and soap as fatty acid - to be incorporated by spray-on rather than via the slurry. The term "detergent base composition" as used herein includes those sprayed-on ingredients.

In the more specific context of a spray-dried densified granular composition prepared by the process of the invention, the term "detergent base composition" indicates the spray-dried powder produced by step (1), plus any "base" ingredients introduced separately prior to or during the densification step (2). Postdosed ingredients, that is to say, ingredients added after densification, do not form part of the detergent base composition.

The base composition of the invention may be formulated entirely from environmentally unobjectionable ingredients while exhibiting efficient cleaning performance. Preferred compositions of the invention are substantially free of non-soap anionic surfactants, and yet exhibit cleaning performance comparable with that of conventional detergent powders on the market today based on sulphonate- or sulphate-type anionic surfactants.

The base composition of the invention is also preferably free of phosphate builders. Efficient detergency building is provided by alkali metal aluminosilicate, in a relatively large amount, assisted by an organic sequestrant which is advantageously an environmentally preferred material free of nitrogen or phosphorus, in particular a non-polymeric polycarboxylate such as citrate.

Ingredients which have been the subject of possible objections on environmental grounds, and which are preferably substantially absent, are sodium sulphate, fluorescer, nitrogen- or phosphorus-containing sequestrants, and acrylic, maleic or acrylic/maleic polymers. The base composition of the invention is capable of providing very acceptable cleaning performance without the need for the inclusion of those ingredients.

The composition has a bulk density of at least 650 g/litre and if desired may be compacted to bulk densities of 700 g/litre and above, and even to 750 g/litre and above, the compactness providing a further environmental benefit of reduced packaging requirements. Powder properties (flow, compressibility, resistance to caking) are good whether or not the composition is densified.

Most surprisingly, densification does not have a detrimental effect on the ability of the composition to dispense from the drawer of a drum-type automatic washing machine. This behaviour is in marked contrast to that of conventional powders based on sulphonate- or sulphate type anionic surfactants; in general the dispensing of densified powders of conventional formulation is so poor that dosing via the dispenser drawer is not practicable and the manufacturer has to provide a special reusable dosing device or "shuttle". This is not universally acceptable to the consumer, and being generally of plastics material adds to the waste disposal problem.

Densified detergent base powder of the invention produced by one specific process, described in more detail below, is also characterised by a large and uniform particle size and a regular (spherical) particle shape, giving it an especially attractive appearance, and reducing the amount of further handling (for example, sieving) required after densification.

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The fatty acid soap

The base composition of the invention contains from 5 to 40 wt%, preferably from 5 to 30 wt%, more preferably from 5 to 20 wt% and desirably from 7 to 20 wt%, of fatty acid soap. The soap provides detergency, detergency building and foam control.

This total will generally be a blend composed of soaps of different chain lengths, and of saturated and unsaturated soaps. Commercially available soaps suitable for use in the present invention include those derived from hardened or natural tallow, coconut oil, rapeseed oil, and mixtures of these. It is also possible, however, to manipulate the chain length distribution and the amount of unsaturation so as to maximise the efficiency of the final blend as surfactant and (to a lesser extent) as builder, while giving also giving optimum stability, and dissolution and dispersion properties.

The soap may advantageously contain at least 40 wt% unsaturated soap. This is preferably in the form of oleate (singly unsaturated soap) rather than linoleate (doubly unsaturated soap) because the latter material can have a tendency to rancidity.

If the composition is intended particularly for use in low-temperature washing, a soap blend is advantageously chosen that has a Krafft point not higher than 50°C, and preferably not higher than 30°C, so that optimum dispersion of the soap throughout the wash liquor and dissolution during the wash cycle is ensured.

For example, GB 2 034 741B (Unilever) discloses a soap blend having a low Krafft temperature (below 25°C), derived from a C₁₂₋₂₂ fatty acid mixture comprising

- (i) from 5 to 60 wt% of one of more saturated or unsaturated fatty acids having 14 or fewer carbon atoms,
- (ii) from 5 to 32 wt% of one or more saturated fatty acids having more than 14 carbon atoms,
- (iii) from 35 to 90 wt% of one mor more unsaturated fatty acids having 14 or more carbon atoms.

One soap blend suitable for use in the base composition of the invention, derived from tallow and coconut, contains 14 wt% C₁₂ saturated soap, 40 wt% C₁₈ saturated soap, and 46 wt% C₁₈ unsaturated soap (oleate). This blend has a Krafft point of about 45 °C and exhibits excellent dispersion, dissolution and detergency in the 60 °C wash cycle. Another blend, especially suitable for very low temperature washing, contains a higher proportion of short-chain saturated soap (39 wt%), and a lower proportion of longer-chain saturated soap (15 wt%), and has a Krafft point of about 12 °C.

The alkoxylated nonionic surfactant

The base composition of the invention also contains from 5 to 35 wt%, preferably from 5 to 20 wt%, and more preferably from 5 to 15 wt%, of alkoxylated nonionic surfactant.

Nonionic surfactants are well known to those skilled in the art, and include the C_{12-20} primary and secondary aliphatic alcohols ethoxylated with an average of from 3 to 20 moles of ethylene oxide per mole of alcohol. Especially preferred for use in the base composition of the invention are those with lower degrees of ethoxylation, for example, 3 to 8 moles of ethylene oxide per mole of alcohol.

Nonionic surfactants of plant or animal origin, for example, the ethoxylation products of tallow alcohol, hardened tallow alcohol, and coconut alcohol, may if desired be used in the base composition of the present invention. One preferred nonionic surfactant for use in the base composition of the invention is coconut alcohol ethoxylated with an average of from 3 to 8, preferably from 5 to 8, moles of ethylene oxide; optionally in combination with tallow or hardened tallow alcohol ethoxylated with an average of 6 to 10 moles of ethylene oxide.

The alkali metal aluminosilicate

The principal detergency builder of the base composition of the invention is alkali metal aluminosilicate, present in a substantial amount: 20 to 60 wt%, preferably 25 to 60 wt%, more preferably from 30 to 50 wt%, calculated on an anhydrous basis.

The alkali metal (preferably sodium) aluminosilicate builder may be crystalline or amorphous or a mixture thereof, and has the general formula 0.8-1.5 Na₂O.Al₂O₃.0.8-6 SiO₂.

These materials contain some bound water and are required to have a calcium ion exchange capacity of at least about 50 mg CaO/g. The preferred aluminosilicates contain 1.5-3.5 SiO₂ units (in the formula above) and have a particle size of not more than about 100 μm, preferably not more than about 20 μm. Both amorphous and crystalline aluminosilicates can be made readily by reaction between sodium silicate

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and sodium aluminate, as amply described in the literature.

Crystalline aluminosilicates (zeolites) are preferred for use in the present invention. Suitable materials are described, for example, in GB 1 473 201 (Henkel) and GB 1 429 143 (Procter & Gamble). The preferred sodium aluminosilicates of this type are the well-known commercially available zeolites A and X, and mixtures thereof. Especially preferred for use in the present invention is Type 4A zeolite.

The novel zeolite P described and claimed in EP 384 070A (Unilever) published on 29 August 1990 may also be used in the base composition of the invention.

10 The organic sequestrant cobuilder

The aluminosilicate is assisted by an organic sequestrant cobuilder, preferably a material free of nitrogen or phosphorus, present in an amount of from 2 to 15 wt%, preferably from 5 to 10 wt%.

The organic builder is preferably a water-soluble salt of a polycarboxylic acid, for example, alkali metal (preferably sodium) oxydisuccinate, tartrate monosuccinate, tartrate disuccinate, carboxymethyloxysuccinate, polyacetal carboxylate, or, most preferably, citrate. Especially preferred are water-soluble salts of non-polymeric polycarboxylic acids. Water-soluble (preferably alkali metal) salts of citric acid, for example, sodium citrate, are especially preferred. Most preferably, sodium citrate is used in an amount of from 5 to 10 wt%.

Polymeric polycarboxylates, especially acrylic, maleic and acrylic/maleic polymers, are also effective cobuilders and their use is not excluded from the scope of the invention, although the materials listed in the previous paragraph may be preferred on environmental grounds.

25 Sodium carbonate

To provide alkalinity in the wash, sodium carbonate is advantageously present in an amount of up to 25 wt%, for example, from 2 to 25 wt%, preferably from 5 to 15 wt%. If the amount of fatty acid soap is 6 wt% or less, then the presence of 2 to 25 wt% sodium carbonate is an essential feature of the invention.

It is also within the scope of the invention, however, for base compositions containing more than 6 wt% of fatty acid soap to be free of sodium carbonate.

Other optional ingredients

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If desired, a low level of water-soluble alkali metal silicate, preferably sodium silicate not more than 5 wt% - may be included in order to provide protection against corrosion of metal washing machine parts, to increase alkalinity, and to provide powder structure. Preferably the amount of sodium silicate does not exceed 2 wt%.

Powder structuring may also be assisted by the inclusion of succinic acid and/or a water-soluble (preferably alkali metal) salt thereof, more preferably sodium succinate, as described and claimed in EP 61 295B (Unilever). An amount of up to 5 wt% (calculated as succinic acid) is sufficient, and preferably the amount does not exceed 2.5 wt% calculated as succinic acid or 3.5 wt% calculated as sodium succinate.

The base composition of the invention may also contain other conventional minor ingredients. As previously indicated, however, the base composition of the invention preferably does not contain materials which have been the subject of doubts or objections on environmental grounds.

Non-base ingredients

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The base composition of the invention is suitable for use in its own right as a non-bleaching, non-enzymatic detergent composition. If desired, however, further ingredients may be admixed with the base composition to provide additional performance benefits.

Examples of ingredients that may be admixed include peroxy bleach systems, proteolytic enzymes, so lipolytic enzymes, amylolytic enzymes, fabric softening granules, and perfumes.

A suitable peroxy bleach system consists of an inorganic persalt, for example, sodium perborate tetrahydrate, sodium perborate monohydrate or sodium percarbonate, in combination with a bleach precursor, for example, tetraacetylethylenediamine (TAED). If desired, a bleach stabiliser (heavy metal

sequestrant) may also be present, although, as previously indicated, it may be preferred to avoid the use of sequestrants containing nitrogen or phosphorus. The peroxy bleach system is suitably present in an amount of from 5 to 30 wt%, preferably from 5 to 15 wt%.

Preferred fabric softening granules are those comprising clay and nonionic surfactant in a weight ratio of from 2:3 to 20:1, as described and claimed in EP 287 343A and EP 287 344A (Unilever). These are suitably present in an amount such that the amount of clay in the final composition is within the range of from 1.5 to 35 wt%, preferably from 4 to 15 wt%, the nonionic surfactant specifically associated with the clay being accounted for within the total amount of nonionic surfactant already specified.

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Preparation of the detergent base composition

As previously indicated, the detergent base composition of the invention may be prepared by any suitable process, but processes involving spray-drying are of especial interest. For compositions with bulk densities at the lower end of the range, for example, around 650 g/litre, spray-drying may be employed without post-treatment.

However, the preferred process for the preparation of the base composition of the invention consists of two main steps: spray-drying an aqueous slurry to produce a powder of conventional bulk density, followed by densification in a high-speed mixer/granulator.

The soap, aluminosilicate, organic builder, and (if present) sodium carbonate, sodium silicate and sodium succinate, are all suitable for incorporation into the base composition via the slurry, although it may be desirable to incorporate these materials in part at a later stage of the process, before or during densification. The addition of liquid or liquefiable ingredients during densification so that they can act as binders or granulating aids may be beneficial.

Nonionic surfactant may be included in the slurry and/or added at a later stage in the process, for example, sprayed on before or during densification; generally nonionic surfactant having a relatively low degree of ethoxylation is not included in the slurry, but the incorporation of a low level of nonionic surfactant in the slurry aids processing.

Similarly, soap may if desired be incorporated wholly or in part by spray-on of the corresponding fatty acid before or during densification.

It may also be desirable, for ease of processing, to add part of the aluminosilicate, and/or sodium carbonate if present, during densification; or to postdose additional aluminosilicate, sodium carbonate or other inorganic solids after densification.

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The high-speed mixer/granulator

Densification of the base powder is preferably effected by means of a high-speed mixer/granulator having both a stirring action and a cutting action. Preferably the stirrer and the cutter may be operated independently of one another, and at separately variable speeds. Such mixers are capable of combining a high energy stirring input with a cutting action, but can also be used to provide other, gentler stirring regimes with or without the cutter in operation. They are thus highly versatile and flexible pieces of apparatus.

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The bowl-type batch mixer

According to a first preferred embodiment of the process of the invention, the high-speed mixer/granulator is a bowl-shaped batch mixer which more preferably has a substantially vertical stirrer axis. Especially preferred are mixers of the Fukae (Trade Mark) FS-G series manufactured by Fukae Powtech Kogyo Co., Japan; this apparatus is essentially in the form of a bowl-shaped vessel accessible via a top port, provided near its base with a stirrer having a substantially vertical axis, and a cutter positioned on a side wall. The stirrer and cutter may be operated independently of one another, and at separately variable speeds.

Other similar mixers found to be suitable for use in the process of the invention are the Diosna (Trade Mark) V series ex Dierks & Söhne, Germany; and the Pharma Matrix (Trade Mark) ex T K Fielder Ltd., England. Other similar mixers believed to be suitable for use in the process of the invention include the Fuji (Trade Mark) VG-C series ex Fuji Sangyo Co., Japan; and the Roto (Trade Mark) ex Zanchetta & Co srl,

italy

Another mixer found to be suitable for use in the batch process of the invention is the Lödige (Trade Mark) FM series batch mixer ex Morton Machine Co. Ltd., Scotland. This differs from the mixers mentioned above in that its stirrer has a horizontal axis.

Referring now again to the preferred Fukae type of mixer, densification is generally effected by running the mixer at a relatively high speed using both stirrer and cutter; a relatively short residence time (for example, 5-8 minutes for a 35 kg batch) is generally sufficient. The final bulk density can be controlled by choice of residence time, and it has been found that the powder properties (flow, compressibility, resistance to caking) of the resulting granulate are optimum if the bulk density has been allowed to rise to at least 750 g/litre, preferably at least 800 g/litre.

This process produces a dense, granular product of very uniform particle size and very regular, generally spherical particle shape which is exceptionally visually attractive. Generally the average particle size is within the range of from 700 to 800 μ m, the n-value being typically about 2.5; with only about 12 15 wt% oversize (>1000 μ m) material and, more importantly, a very low level (typically only about 2-3 wt%) of "fines" (material <180 μ m). Particle porosity is generally less than 0.25, and preferably less than 0.20.

The presence of a small amount of liquid binder may be required during the densification step. Water may be added for this purpose, or, as previously indicated, part of the nonionic surfactant and/or the soap (as fatty acid) may be withheld from the slurry and incorporated at this stage. The binder may be sprayed in while the mixer is running. If desired, the mixer may first be operated at a relatively slow speed while binder is added, before increasing the speed of the mixer to effect densification.

The optimum granulation temperature in this type of process appears to be formulation-dependent. According to the above-mentioned EP 340 013A (Unilever), the densification process is suitably carried out at a controlled temperature somewhat above ambient, preferably above 30°C, more preferably from 30 to 45°C; however, with the type of formulation with which the present invention is concerned, the best results appear to be obtained when the granulation temperature does not exceed 30°C, and a temperature of about 25°C appears to be particularly favourable. At higher temperatures, a wider particle size distribution may be obtained, and it may be necessary to remove oversize granules and "fines" by sieving.

With this type of formulation, generally containing a relatively high level of aluminosilicate and no anionic surfactant, it has been found that no "agent for improving surface properties" as defined in the above-mentioned JP 61 069 897A (Kao) need be present during densification, although the use of such a material is not excluded from the scope of the invention.

The continuous mixer

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According to a second preferred process of the invention, densification is effected by means of a continuous high-speed mixer/granulator, preferably having a substantially horizontal stirring axis.

The preferred mixer of this type is the Lödige (Trade Mark) CB 30 or CB 100 Recycler. This mixer consists essentially of a large static hollow cylinder with a central rotating shaft (stirring axis) carrying several different types of blades. It can be rotated at speeds of 100-2500 rpm, depending on the degree of densification and the particle size desired. The blades on the shaft provide a thorough mixing action of the solids and any liquids admixed at this stage. The mean residence time is somewhat dependent on the rotational speed of the shaft, the position of the blades and the weir at the exit opening, but in general a relatively short residence time of about 5 to 30 seconds is suitable.

Alternative high-speed mixer/granulators of this general type include the Shugi (Trade Mark) Granulator and the Drais (Trade Mark) K-TPP 80 mixer.

To obtain maximum benefit from treatment in the preferred Lödige Recycler, the particulate starting material (predensified detergent base composition) is preferably brought into, or maintained in, a deformable state. This may be induced in a number of ways, for example, by operating at a temperature of 45°C or above; however, when liquid ingredients such as water or nonionic surfactant are added to the particulate starting material, lower temperatures, for example, 35°C and above, may be employed.

One simple method of ensuring that the particulate starting material is in a deformable state is to feed spray-dried base powder leaving the tower at a temperature of ≥45°C directly into the high-speed mixer/granulator. If plant layout does not permit that, the powder may be cooled first, for example in an airlift, and then reheated after transportation to the high-speed mixer/granulator.

For subsequent handling and use, the densified detergent base composition emerging from the Lödige Recycler should no longer be in a deformable state. The densification step is therefore preferably followed by a drying and/or cooling step. This may be carried out in any suitable manner, for example, using a fluid

bed (drying) or an airlift (cooling).

In view of the short residence time, the amount of densification that can be achieved using the Lödige Recycler is generally rather less than that achievable using the batch process previously described. Bulk densities of 650-700 g/litre are however readily obtained.

It may therefore be desirable, when operating continuously, to subject the densified composition to a further processing step: treatment in a moderate-speed mixer/granulator, suitably with a residence time of form 1 to 10 minutes, preferably from 2 to 5 minutes. As with the first densification step, the composition should be in a deformable state, so any drying and/or cooling should be carried out after this further step.

This further step may be carried out in a Lödige KM 300 mixer, also known as the Lödige Ploughshare. This apparatus consists essentially of a hollow static cylinder having a central rotating shaft (stirring axis), on which various plough-shaped blades are mounted. The shaft can be rotated at speeds of from 40 to 160 rpm; optionally, one or more high-speed cutters may be used to prevent excessive agglomeration.

Another mixer suitable for this process step is the Drais K-T 160.

Using this combined process, bulk densities as high as 960 g/litre have been achieved.

A continuous process for the production of a high bulk density detergent powder, comprising the sequential use of a high-speed mixer/granulator and a moderate-speed mixer/granulator, followed by a drying and/or cooling step, is described and claimed in EP 367 339A (Unilever) published on 9 May 1990.

If desired, small amounts of finely powdered solids, for example, sodium aluminosilicate, may be added in the moderate-speed mixer/granulator and/or the high-speed mixer/granulator, as described in EP 390 251A (Unilever) published on 3 October 1990.

Powder properties and dispensing

The processes of the invention generally produce densified detergent base compositions having excellent flow properties.

For products of the batch process, the dynamic flow rate typically exceeds 120 ml/s, and compressibility is less than 15% v/v. Surprisingly, the batch densification process generally produces an improvement in powder properties.

The continuous process using only the high-speed mixer/granulator (Lödige Recycler) typically gives products having dynamic flow rates of 100 ml/s or above, and compressibilities of less than 15% v/v.

The products of both batch and continuous processes have also been found to dispense rapidly and efficiently in drum-type automatic washing machines, typically leaving residues of 2 wt% or less in the test described below.

The dispensing properties of compositions of the invention in a drum-type front-loading automatic washing machine were determined by means of a standard test using a Philips (Trade Mark) AWB 126/7 washing machine using a 100 g powder dose and a water fill of 5 litres at 10°C or 20°C flowing in at a pressure of 0.5 bar, over a period of 1 minute. The powder remaining in the dispenser after that 1-minute period was removed, dried at 100°C for 12 hours, and weighed. This weight, in grams, represented the weight percentage of powder not dispensed into the machine (the residue). An average of at least four determinations was taken as the final result. It will be appreciated that this test is stringent, using a low water inlet temperature and flow rate, and a machine with a drawer-type dispenser that is especially vulnerable to high residues and clogging.

EXAMPLES

The invention is further illustrated by the following non-limiting Examples.

Example 1, Comparative Examples A and B

A granular detergent base composition was prepared to the following formulation:

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	% (final)	% (base)
Sodium soap (46 wt% unsaturated)	13.10	13.26
Nonionic surfactant:		_
Tallow alcohol 8EO	3.75	3.80
Coconut alcohol 6.5EO	5.00	5.06
Zeolite 4A (anhydrous basis)	43.80	44.34
Sodium citrate	6.25	6.33
Sodium carbonate	6.25	6.33
Sodium succinate	2.61	2.64
Sodium silicate	0.90	0.91
Moisture	17.12	17.33
	98.78	100.00

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The soap blend used was the sodium salt of a fatty acid mix composed of 14 wt% lauric acid, 40 wt% stearic acid, and 46 wt% oleic acid.

The tallow alcohol 8EO, soap (as fatty acid), zeolite, sodium citrate, sodium carbonate, sodium succinate (as succinic acid) and sodium silicate were slurried with water and spray-dried to form a powder having a moisture content of about 18 wt%, including the water of hydration of the zeolite; this represents a free moisture content of about 7 wt%. The coconut alcohol 6.5EO was then sprayed on. The bulk density was about 300-550 g/litre.

A 50 kg batch of this powder was then densified in a Fukae FS-100 high-speed mixer/granulator. The stirrer speed was 130 rpm, the cutter speed was 2000 rpm and the residence time was 10 minutes. The resulting densified granulate had a bulk density of about 830 g/litre. It was free-flowing and showed no tendency to cake. It was of very attractive appearance, being composed of relatively large substantially spherical granules with a very narrow particle size distribution.

Postdosed ingredients were added as follows:

	% (final)
Enzyme (savinase) granules	1.00
Perfume	0.22
	100.00

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The enzyme granules were admixed with the densified granulate, and the perfume then sprayed on, to give an enzymatic detergent composition (Composition 1).

For comparison (Comparative Composition A), the same postdosed ingredients were admixed in the same proportions with an undensified sample of the base powder.

The powder properties of the two compositions, and those of a sample (Comparative Composition B) of a high bulk density detergent powder commercially available in the United Kingdom and containing 9 wt% of non-soap anionic surfactants, 8 wt% nonionic surfactant, 0.5 wt% soap and 18.5 wt% zeolite, were as follows:

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	1	A	В
Bulk density (g/l)	827	550	763
Dynamic flow rate (ml/s)	142	80	63
Average particle size (µm)	768	500	407
Particle size distribution n	2.6	1.9	1.6
Fines (<180 µm)	2.2	16	23
Compressibility (v/v)	12	16	13

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Dispenser residues (each the mean of 7 runs) were as follows:

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	1	A	В
Residue at 20 °C (wt%) Residue at 10 °C (wt%)	1.1	1.2	70
	1.2	2.4	70

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Example 2

A densified base powder of the formulation given in Example 1 was prepared using a slightly different method according to which the coconut alcohol 6.5EO was sprayed into the Fukae mixer during the densification step rather than sprayed onto the powder prior to densification. A similar product, of very attractive appearance, was obtained.

The densified base powder had the following properties:

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2.5
13
<1

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Examples 3 to 7

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Base powders were prepared to the formulations shown in Table 1, and were densified, as described in Example 1, using the Fukae mixer.

Powder properties were as shown in Table 2. All the densified powders were extremely attractive in appearance.

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Table 1

Examples 3 to 7 - Base powder compositions Example 3 Example 4 Example 5 Example 6 Example 7 parts parts parts parts parts Sodium soap*: Blend A 13.10 15.25 15.25 Biend B 13.10 17.50 Nonionic surfactant: Tallow 8EO (slurry) 10.25 10.25 Coconut 6.5EO (slurry) 3.75 3.75 3.60 Coconut 6.5EO (sprayed) 5.00 5.00 8.00 Zeolite 4A (anhydr) 43.80 43.80 40.50 40.50 33.50 Sodium citrate 6.25 7.25 7.25 6.25 8.50 Sodium carbonate 6.25 6.25 7.25 7.25 8.50 Sodium succinate 2.61 2.61 2.61 Sodium silicate 0.90 0.90 1.00 1.00 1.20 Moisture 18.00 18.00 17.00 17.00 18.11 99.66 99.66 98.50 98.91 101.11

Table 2

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Examples 3 to 7 (continued)						
Properties of densified base powders						
Example 3 4 5 6 7						
Bulk density (g/l) Dynamic flow rate (ml/s) Compressibility (%v/v) Average particle size (µm) Particle size distribution n Fines (wt% <180 µm)	808 123 14 728 1.7 5.6	854 141 11 831 1.9 5.0	810 140 12 785 1.9 3.5	822 138 11 746 2.1 3.1	840 142 13 900 2.3 1.8	
Dispenser residues 20 °C	all below 1 wt%					
10°C	all below 1 wt%					

Example 8, Comparative Example C

A spray-dried powder was prepared to the following formulation:

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^{*} Blend A: Na salt of 39% lauric/15% stearic/46% oleic acid mix Blend B: Na salt of 14% lauric/40% stearic/46% oleic acid mix

% (base) parts Sodium soap (as Example 1) 13.00 13.77 Nonionic surfactant: Tallow alcohol 8EO 3.80 4.03 Zeolite 4A (anhydrous basis) 44.00 46.61 Sodium citrate 6.30 6.67 Sodium carbonate 6.30 6.67 Sodium succinate 2.61 2.76 Sodium silicate 0.90 0.95 Minor ingredients 0.50 0.53 Water 12.00 12.71 89.41 94.70

The spray-dried powder was fed directly into a Lödige Recycler CB 30 continuous high-speed mixer/granulator, described in more detail previously. The mixer speed was 180 rpm, the mixer tip speed 8-30 m/s, the residence time 1-2 minutes, and the throughput 0.9 t/hr. In the Recycler, the following ingredient was added:

Nonionic surfactant:		
Coconut alcohol 6.5EO	5.00	5.30
	94.51	100.00

A cooling/drying step was then carried out in a fluid bed.

The properties of the densified base powder leaving the Lödige Recycler (Example 8a), on leaving the fluid bed (Example 8b) and of a sample of the spray-dried powder prior to densification (Comparative Example C) were as follows:

	<u>c</u>	8a	8b
Bulk density (g/l) Average particle size (μm) Moisture content (wt%)	360	738	755
	460	730	740
	11.9	11.8	11.2

Finally, postdosed ingredients were added to the densified base composition of Example 8 as follows:

Enzyme	0.50
Perfume	0.30
	95.31

Properties of the final product were as follows:

Bulk density (g/l)	680
Average particle size (µm)	690
Dynamic flow rate (ml/s)	84
Dispenser residues (10°C, 20°C)	0

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Examples 9 to 12

A base powder of the formulation given in Example 1 was densified continuously using a Lödige Recycler as described previously, and the densified product emerging from the Recycler was sampled at four different times.

The samples (listed in chronological order of sampling) had the following bulk densities and gave the following dispenser residues at 10° C:

	9	10	11	12
Bulk density (g/l)	705	710	703	686
Dispenser residues (wt %)	<1	<1	<1	<1

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Example 13

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This Example describes a light-duty bleaching enzymatic detergent composition in accordance with the present invention.

The detergent base composition was that of Example 1, and was densified in the Fukae mixer as described in that Example:

	% (final)	% (base)
Sodium soap	11.98	13.26
Nonionic surfactant:		
Tallow alcohol 8EO	3.43	3.80
Coconut alcohol 6.5EO	4.57	5.06
Zeolite 4A (anhydrous)	40.05	44.34
Sodium citrate	5.71	6.33
Sodium carbonate	5.71	6.33
Sodium succinate	2.39	2.64
Sodium silicate	0.82	0.91
Moisture	15.66	17.33
	90.32	100.00

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Postdosed ingredients were added as follows:

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	% (final)
Enzyme (Maxatase) granules	0.40
Perfume	0.28
Sodium perborate monohydrate	6.00
TAED granules	3.00
	100.00

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The sodium perborate monohydrate, TAED granules and enzyme granules were admixed with the densified granulate, and the perfume sprayed on, to give a high-bulk-density enzymatic bleaching detergent composition.

Powder properties of the final product were as follows:

Bulk density (g/l)	780-800
Dynamic flow rate (ml/s)	140
Compressibility (% v/v)	5
Dispenser residue (wt%) (20°C)	<1

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Example 14

A composition similar to that of Example 13 but intended for heavy duty laundry use could be formulated as follows:

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Example 15, Comparative Examples D, E and F

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The composition of this Example contained fabric softening granules of clay carrying adsorbed nonionic surfactant (3EO).

Base powder

		<u>\$</u>	<u>ड</u>
		(final)	(base)
. 40			
	Sodium soap (as Example 1)	9.89	14.19
	Nonionic surfactant:		
45	Tallow alcohol 8EO	2.42	3.47
75	Coconut alcohol 6.5E0*	3.55	5.09
	Zeolite 4A (anhydrous)	29.92	42.91
	Sodium citrate (as dihydrate)	4.72	6.77
50	Sodium carbonate	3.02	4.33
	Sodium succinate	1.78	2.55
	Moisture	14.42	20.69
55			
	*sprayed on	69.72	100.00

Postdosed

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		<pre>% (final)</pre>
5		
	Sodium bicarbonate	1.28
	Sodium perborate monohydrate	12.00
	TAED	5.00
10	Enzyme (Savinase 6.0T)	0.80
	Clay/nonionic granules	11.20
15		100.00

The detergency of the product of Example 15 in a 60 °C machine wash on various test cloths carrying standard soils was compared with those of three products (Comparative Examples D, E and F) containing anionic non-soap surfactants. The formulations of the three comparative products are shown below.

	D	E	F
		- (% final)	•
Linear alkylbenzene sulphonate	5.40	6.02	6.73
Nonionic surfactant:			
C ₁₂₋₁₅ 7EO	2.47	2.76	2.68
C12-15 3EO	2.13	6.00	6.71
Sodium soap	1.50	1.80	1.85
Zeolite 4A (anhydrous)	21.58	24.06	26.94
Acrylic/maleic copolymer	2.43	3.61	4.04
Sodium silicate	0.36	0.40	0.45
Sodium carbonate	12.56	12.20	11.35
Sodium sulphate	15.71	-	-
Na carboxymethylcellulose	0.45	0.50	0.56
Fluorescers	0.17	0.19	0.21
Antifoam granules	0.50	1.56	1.20
Sodium perborate:			
tetrahydrate	11.36	-	-
monohydrate	-	13.00	14.00
TAED	- '	3.00	7.35
Dequest	•	-	0.75
Enzyme (Savinase 6.0T)	0.33	0.58	1.10
Clay/nonionic granules	10.00	11.20	-
Perfume	0.32	0.40	0.35
Moisture, minor ingredients	12.73	13.42	13.73
	100.00	100.00	100.00

The results, shown as reflectance increases at 460 nm, were as follows (C = cotton, P/C = polyester/cotton):

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Soil/Fabric	15	D	E	F
oil/silica/ink, C	26.4	25.2	26.3	27.2
casein, C	33.6	32.3	33.3	33.9
indian ink/olive oil, C	22.9	20.3	18.1	22.7
indian ink/olive oil, P/C	29.7	27.9	26.4	25.6

Stain removal results (percentage stain removal) in the 60 °C wash were as follows:

Stain	15	D	E	F
Blackberry	5.0	4.0	2.5	1.7
Blood	3.6	4.5	2.7	1.6
Gravy	3.9	4.1	3.4	2.3

It is evident that the product of Example 15 generally performed at least as well as the comparative products.

Examples 16 to 21

In Table 3 there are shown some further formulations according to the invention, containing different nonionic surfactants, the Synperonic (Trade Mark) series ex ICI. Amounts shown are in parts by weight.

Examples 22 to 27

In Table 4 there are shown some further formulations according to the invention, similar to those of Examples 16 to 21 but containing acrylic/maleic copolymer (Sokalan (Trade Mark) CP5 ex BASF) instead of sodium citrate.

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Table 3

Examples 16 to 21						
	16	<u>17</u>	18	19	20	21
Base composition						
Soap (as Ex 1)	7.2	7.2	7.2	6.3	6.3	6.3
Nonionic surfactdant:	_					
C ₁₂₋₁₅ 7EO (slurry)	6.2	6.2	6.2	5.4	5.4	5.4
C ₁₂₋₁₅ 3EO (spray-on)	5.8	5.8	5.8	6.3*	6.3*	6.3*
Zeolite 4A (anhydrous)	32.3	32.3	32.3	27.9	27.9	27.9
Sodium citrate (dihydrate)	4.6	4.6	4.6	4.0	4.0	4.0
Sodium carbonate	-	9.8	•	-	8.5	8.5
Sodium carboxymethyl cellulose	0.5	0.5	0.5	0.5	0.5	0.5
Fluorescers	0.2	0.2	0.2	0.2	0.2	0.2
Moisture	16.1	16.1	16.1	14.0	14.0	14.0
Postdosed						
Sodium perborate monohydrate	13.0	13.0	13.0	13.0	13.0	13.0
TAED	5.0	5.0	5.0	5.0	5.0	5.0
Enzyme (Savinase)	0.6	0.6	0.6	0.6	0.6	0.6
Perfume	0.25	0.25	0.25	0.25	0.25	0.25
Clay/nonionic granules (as clay)	-	-	-	9.2	9.2	9.2
Sodium carbonate	9.8	-	9.8	8.5	•	8.5
Sodium sulphate	-	-	20.0	•	-	20.0

*includes nonionic surfactant present in clay/nonionic granules

Table 4

	Example	es 22 to 2	7			
	22	23	24	25	26	27
Base composition				_	_	
Soap (as Ex 1)	7.2	7.2	7.2	6.3	6.3	6.3
Nonionic surfactant:	1					
C ₁₂₋₁₅ 7EO (slurry)	6.2	6.2	6.2	5.4	5.4	5.4
C ₁₂₋₁₅ 3EO (spray-on)	5.8	5.8	5.8	6.3*	6.3*	6.3*
Zeolite 4A (anhydrous)	32.3	32.3	32.3	27.9	27.9	27.9
Acrylic/maleic copolymer	4.6	4.6	4.6	4.0	4.0	4.0
Sodium carbonate	-	9.8	-	-	8.5	8.5
Sodium carboxymethyl cellulose	0.5	0.5	0.5	0.5	0.5	0.5
Fluorescers	0.2	0.2	0.2	0.2	0.2	0.2
Moisture	16.1	16.1	16.1	14.0	14.0	14.0
Postdosed						
Sodium perborate monohydrate	13.0	13.0	13.0	13.0	13.0	13.0
TAED	5.0	5.0	5.0	5.0	5.0	5.0
Enzyme (Savinase)	0.6	0.6	0.6	0.6	0.6	0.6
Perfume	0.25	0.25	0.25	0.25	0.25	0.25
Clay/nonionic granules (as clay)		-	-	9.2	9.2	9.2
Sodium carbonate	9.8	-	9.8	8.5	-	8.5
Sodium sulphate	-	-	20.0	-	-	20.0

"includes nonionic surfactant present in clay/nonionic granules

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Claims

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- 1. A granular detergent base composition having a bulk density of at least 650 g/litre, which comprises:
- (i) an organic surfactant system consisting essentially of:
 - (a) from 5 to 40 wt% of fatty acid soap,
 - (b) from 5 to 35 wt% of alkoxylated nonionic surfactant; (ii) a detergency builder system consisting essentially of:
 - (c) from 20 to 60 wt% (anhydrous basis) of crystalline or amorphous alkali metal aluminosilicates
 - (d) from 2 to 15 wt% of an organic sequestrant builder; (iii) optionally from 0 to 25 wt% sodium carbonate, with the proviso that if the content of fatty acid soap is ≤6 wt%, from 2 to 25 wt% sodium carbonate is present;
- 45 (iv) optionally other detergent ingredients and moisture 30 to 100 wt%,
 - all percentages being based on the detergent base composition.
 - 2. A detergent base composition as claimed in claim 1, characterised in that it is substantially free of anionic non-soap surfactant.
- 3. A detergent base composition as claimed in claim 1 or claim 2, characterised in that it comprises from 5 to wt% of fatty acid soap (a).
 - 4. A detergent base composition as claimed in claim 3, characterised in that it comprises from 5 to 20 wt% of fatty acid soap (a).
 - 5. A detergent base composition as claimed in claim 4, characterised in that it comprises from 7 to 20 wt% of fatty acid soap (a).
- 6. A detergent base composition as claimed in any preceding claim, characterised in that the fatty acid soap (a) comprises a mixture of saturated and unsaturated soap.
 - 7. A detergent base composition as claimed in claim 6, characterised in that the fatty acid soap (a) contains at least 40 wt% unsaturated soap.

- 8. A detergent base composition as claimed in any preceding claim, characterised in that the fatty acid soap has a Krafft point not higher than 50°C.
- 9. A detergent base composition as claimed in claim 8, characterised in that the fatty acid soap has a Krafft point not higher than 30° C.
- 5 10. A detergent base composition as claimed in any preceding claim, characterised in that it comprises from 5 to 20 wt% of nonionic surfactant (b).
 - 11. A detergent base composition as claimed in claim 10, characterised in that it comprises from 5 to 15 wt% of nonionic surfactant (b).
- 12. A detergent base composition as claimed in any preceding claim, characterised in that the alkoxylated nonionic surfactant (b) comprises a C₁₂₋₂₀ aliphatic alcohol ethoxylated with an average of from 3 to 20 moles of ethylene oxide per mole of alcohol.
 - 13. A detergent base composition as claimed in claim 12, characterised in that the alkoxylated nonionic surfactant (b) comprises a C_{12-20} aliphatic alcohol ethoxylated with an average of from 3 to 8 moles of ethylene oxide per mole of alcohol.
- 15 14. A detergent base composition as claimed in any preceding claim, characterised in that the alkoxylated nonionic surfactant (b) is selected from ethoxylation products of tallow alcohol, hardened tallow alcohol, and coconut alcohol.
- 15. A detergent base composition as claimed in claim 14, which includes as alkoxylated nonionic surfactant
 (b) coconut alcohol ethoxylated with an average of 3 to 8 moles of ethylene oxide, optionally in combination
 with tallow or hardened tallow alcohol ethoxylated with an average of 6 to 10 moles of ethylene oxide.
 - 16. A detergent base composition as claimed in any preceding claim, characterised in that it comprises from 25 to 60 wt% of alkali metal aluminosilicate (c).
 - 17. A detergent base composition as claimed in claim 16, characterised in that it comprises from 30 to 50 wt% of alkali metal aluminosilicate (c).
- 18. A detergent base composition as claimed in any preceding claim, characterised in that it comprises from 5 to 10 wt% of organic sequestrant builder (d).
 - 19. A detergent base composition as claimed in any preceding claim, characterised in that the organic sequestrant builder is a water-soluble salt of a non-polymeric polycarboxylate.
 - 20. A detergent base composition as claimed in claim 19, characterised in that the organic sequestrant builder is a water-soluble salt of citric acid.
 - 21. A detergent base composition as claimed in any preceding claim, characterised in that it comprises from 2 to 25 wt% of sodium carbonate (iii).
 - 22. A detergent base composition as claimed in claim 21, characterised in that it comprises from 5 to 15 wt% of sodium carbonate (iii).
- 35 23. A detergent base composition as claimed in any one of claims 1 to 20, characterised in that it is substantially free of sodium carbonate.
 - 24. A detergent base composition as claimed in any preceding claim, characterised in that it further comprises up to 5 wt% of water-soluble alkali metal silicate.
 - 25. A detergent base composition as claimed in any preceding claim, characterised in that it further comprises up to 5 wt% of succinic acid and/or a water-soluble salt thereof.
 - 26. A detergent base composition as claimed in any preceding claim, characterised in that it is substantially free of sodium sulphate.
 - 27. A detergent base composition as claimed in any preceding claim, characterised in that it is substantially free of fluorescer.
- 28. A detergent base composition as claimed in any preceding claim, characterised in that it is substantially free of phosphorus- or nitrogen-containing sequestrants.
 - 29. A detergent base composition as claimed in any preceding claim, characterised in that it is substantially free of acrylic or acrylic/maleic polymers.
- 30. A detergent base composition as claimed in any preceding claim, characterised in that it has a bulk density of at least 700 g/litre.
 - 31. A detergent base composition as claimed in claim 30, characterised in that it has a bulk density of at least 750 g/litre.
 - 32. A granular detergent base composition having a bulk density of at least 750 g/litre, characterised in that it consists essentially of:
- 55 (a) from 5 to 20 wt% of fatty acid soap,
 - (b) from 5 to 20 wt% of alkoxylated nonionic surfactant;
 - (c) from 25 to 60 wt% (anhydrous basis) of crystalline or amorphous alkali metal aluminosilicate,
 - (d) from 2 to 15 wt% of an organic sequestrant builder;

- (e) from 5 to 15 wt% of sodium carbonate,
- (f) optionally from 0 to 5 wt% of sodium silicate,
- (g) optionally from 0 to 5 wt% of sodium succinate,
- and optionally other detergent ingredients and moisture to 100 wt%, all percentages being based on the detergent base composition.
 - 33. A process for the preparation of a granular detergent base composition as claimed in any preceding claim, characterised in that it includes the step of spray-drying an aqueous slurry.
 - 34. A process as claimed in claim 33, characterised in that it includes the steps of:
 - (1) spray-drying an aqueous slurry to form a base powder, and
- (2) densifying the spray-dried base powder in a high-speed mixer/granulator having both a stirring action and a cutting action.
- 35. A process as claimed in claim 34, characterised in that part or the whole of the fatty acid soap (a) is incorporated by spraying fatty acid onto the spray-dried base powder before or during the densification step (2).
- 36. A process as claimed in claim 34 or claim 35, characterised in that part or the whole of the nonionic surfactant (b) is sprayed onto the spray-dried base powder before or during the densification step (2).
 - 37. A process as claimed in any one of claims 34 to 36, characterised in that the densification step (2) is carried out in a bowl-type batch high-speed mixer/granulator having a substantially vertical stirrer axis.
 - 38. A process as claimed in any one of claims 34 to 36, characterised in that the densification step (2) is carried out in a continuous high-speed mixer/granulator having a substantially horizontal stirrer axis.
 - 39. A process as claimed in claim 38, characterised in that the densification step (2) is followed by a further densification step (3) carried out in a moderate-speed mixer/granulator.
 - 40. A granular detergent composition, characterised in that it comprises a granular detergent base composition as claimed in any one of claims 1 to 33 in admixture with one or more further detergent ingredients selected from peroxy bleach systems, proteolytic enzymes, lipolytic enzymes, amylolytic enzymes, fabric softening granules, and perfumes.
 - 41. A detergent composition as claimed in claim 40, characterised in that it comprises from 5 to 30 wt% of a peroxy bleach system comprising an inorganic persalt selected from sodium perborate monohydrate, sodium perborate tetrahydrate and sodium percarbonate, in conjunction with a bleach precursor.
- 42. A detergent composition as claimed in claim 40 or claim 41, characterised in that it comprises fabric softening granules comprising clay and nonionic surfactant in a weight ratio of from 2:3 to 20:1, in an amount such that the amount of clay in the detergent composition is within the range of from 1.5 to 35 wt%.

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Applicant: UNILEVER PLC Unilever House Blackfriars P.O. Box 68 London EC4P 4BQ(GB)

Applicant: UNILEVER NV
Burgemeester s'Jacobplein 1 P.O. Box 760
NL-3000 DK Rotterdam(NL)

(M) CH DE ES FR IT LI NL SE

Inventor: Barber, Alan Dan, Unilever Research Port Sunlight Lab., Quarry Road East, Bebington Wirral, Merseyside, L63 3JW(GB) Inventor: Emery, William Derek, Unilever Research Port Sunlight Lab., Quarry Road East, Bebington Wirral, Merseyside, L63 3JW(GB)

Representative: Fransella, Mary Evelyn et al Unilever PLC Patents Division P.O. Box 68 Unilever House London EC4P 4BQ(GB)

(54) Detergent compositions.

A granular detergent base composition having a bulk density of at least 650 g/litre comprises: an organic surfactant system consisting essentially of fatty acid soap (5-40 wt% on composition), alkoxylated nonionic surfactant (5-35 wt% on composition) and preferably free of nonsoap anionic surfactant; a detergency builder system consisting essentially of alkali metal aluminosilicate (20-60 wt% anhydrous on composition) and an organic sequestrant builder such as alkali metal citrate (2-15 wt% on composition);

and optionally various other detergent ingredients such as sodium carbonate, sodium silicate, sodium succinate.

The detergent base composition can be prepared by densification of a spray-dried base powder in a high-speed mixer/granulator. It combines good detergency with a lack of environmentally questionable ingredients, and dispenses well in a drum-type washing machine despite the high bulk density.

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EUROPEAN SEARCH REPORT

EP 90 31 1672

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X	EP-A-0 117 568 (UNILEVI * Whole document *	ER)	24 40	20,23, 1,26-29,) 34	C 11 D 17/06 C 11 D 10/04 C 11 D 3/12
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Υ	EP-A-0 229 671 (KAO CO * Page 36, lines 1-12; claim		1,3 38	34,37, i	
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Y: p C A: t O: r	CATEGORY OF CITED DOCU particularly relevant if taken alone particularly relevant if combined with document of the same catagory echnological background non-written disclosure ntermediate document	MENTS	the filing of D: document L: document	late cited in the cited for of	ent, but published on, or after a application ther reasons